

A molybdenum complex with 4,7-diphenyl-1,10-phenanthroline

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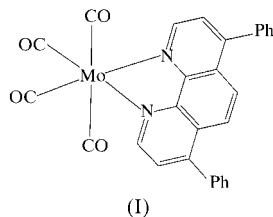
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In the title compound, tetracarbonyl(4,7-diphenyl-1,10-phenanthroline-*N,N'*)molybdenum(0), $[\text{Mo}(\text{C}_{24}\text{H}_{16}\text{N}_2)(\text{CO})_4]$, the Mo-atom coordination is distorted octahedral, with two CO groups *cis* to each other, but each *trans* to an N atom of the 4,7-diphenyl-1,10-phenanthroline (dpphen) ligand, and with the other two CO groups *trans* to each other and on the axis position. The complex has better solubility than $[\text{Mo}(\text{phen})(\text{CO})_4]$, where phen is 1,10-phenanthroline.

Comment

As a precursor, $[\text{M}(\text{L-L})(\text{CO})_4]$ [$\text{M} = \text{Mo}$ or W , $\text{L-L} = 1,10$ -phenanthroline (phen) or 2,2'-bipyridine (bipy); Stiddard, 1962] was reacted with fullerene to form stable molybdenum or tungsten complexes of fullerene $[\text{M}(\eta^2\text{-C}_{60})(\text{L-L})(\text{CO})_3]$ (Tang *et al.*, 1996). These complexes are only sparingly soluble in chlorobenzene and *o*-dichlorobenzene, and crystals suitable for X-ray diffraction have not been obtained. However, when one carbonyl group of $[\text{M}(\eta^2\text{-C}_{60})(\text{phen})(\text{CO})_3]$ was displaced by dibutyl maleate (dbm), the resulting $[\text{M}(\eta^2\text{-C}_{60})(\text{phen})(\text{dbm})(\text{CO})_2]$ complexes have good solubilities in a number of organic solvents and their crystal structures were determined



by X-ray diffraction (Tang *et al.*, 1997). If dbm is replaced by another C_{60} ligand, we can obtain dumb-bell $[\text{M}(\eta^2\text{-C}_{60})_2(\text{L-L})(\text{CO})_2]$ complexes, which are expected to have better optical or electric properties than $[\text{M}(\eta^2\text{-C}_{60})(\text{phen})(\text{dbm})(\text{CO})_2]$ (Zhang *et al.*, 1998). But the key problem is still the poor solubility of the dumb-bell complex, making them difficult to analyse and use in applications. However, when two phenyl groups were introduced into 1,10-phenanthroline, we obtained the title complex, (I), which has much better solu-

bility in aromatic solvents compared with $[\text{Mo}(\text{phen})(\text{CO})_4]$. It could be the precursor for further reaction with fullerenes to obtain complexes which are expected to have good solubility.

The structure determination of (I) indicated that the Mo-atom coordination is distorted octahedral, with two CO groups *cis* to each other, but each *trans* to an N atom of dpphen (Fig. 1). The C2-Mo1-N2 and C3-Mo1-N1 bond angles are $170.52(87)$ and $170.97(7)^\circ$, respectively. Atoms C2, C3, N1, N2 and Mo1 are in the equatorial plane, coplanar to within 0.07 \AA . The dihedral angle between the phen and C2/C3/N1/N2/Mo1 planes is $10.70(8)^\circ$ and the two phenyl groups make angles of $42.45(7)$ and $54.05(5)^\circ$ with the phen plane. The other two CO groups are *trans* to each other and on the axis position of the distorted octahedron.

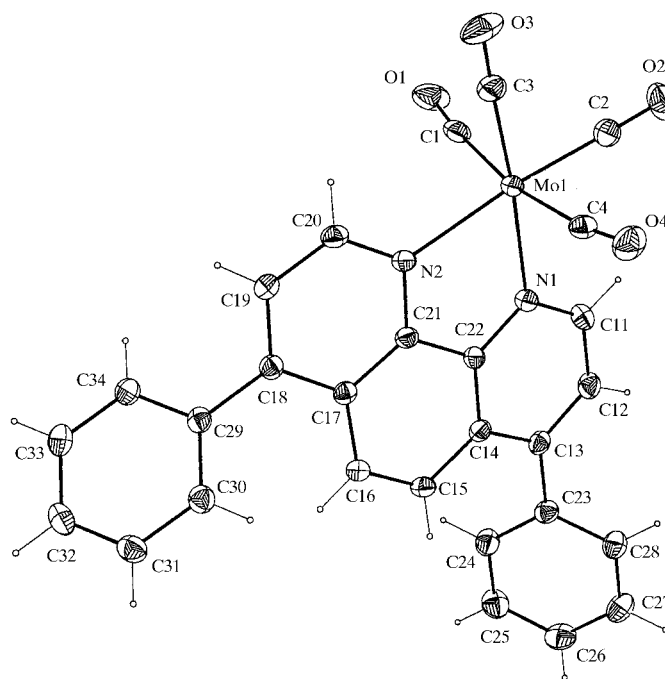


Figure 1
The molecular structure of (I) shown with 50% probability displacement ellipsoids.

Experimental

The title compound was prepared by the reaction of $\text{Mo}(\text{CO})_6$ and dpphen (in a 1:1 molar ratio) in toluene under a nitrogen atmosphere using Schlenk techniques, followed by chromatography over silica gel. Red crystals were formed by diffusion of *n*-pentane into a benzene solution of the title complex.

Crystal data

$[\text{Mo}(\text{C}_{24}\text{H}_{16}\text{N}_2)(\text{CO})_4]$
 $M_r = 540.37$
Monoclinic, $P2_1/c$
 $a = 9.8354(1) \text{ \AA}$
 $b = 7.5648(1) \text{ \AA}$
 $c = 31.1172(4) \text{ \AA}$
 $\beta = 94.4628(4)^\circ$
 $V = 2308.19(5) \text{ \AA}^3$
 $Z = 4$

$D_x = 1.555 \text{ Mg m}^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 39 842 reflections
 $\theta = 2.4\text{--}27.5^\circ$
 $\mu = 0.61 \text{ mm}^{-1}$
 $T = 123(2) \text{ K}$
Block, red
 $0.60 \times 0.40 \times 0.20 \text{ mm}$

Data collection

Rigaku R-AXIS RAPID IP diffractometer	5265 independent reflections
Oscillation scans	4055 reflections with $I > 2\sigma(I)$
Absorption correction: empirical (<i>ABSCOR</i> ; Higashi, 1995)	$R_{\text{int}} = 0.038$
$T_{\text{min}} = 0.662$, $T_{\text{max}} = 0.807$	$\theta_{\text{max}} = 27.5^\circ$
39 842 measured reflections	$h = -12 \rightarrow 12$
	$k = -9 \rightarrow 9$
	$l = -40 \rightarrow 40$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0329P)^2 + 1.7259P]$
$R[F^2 > 2\sigma(F^2)] = 0.023$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.054$	$(\Delta/\sigma)_{\text{max}} = 0.012$
$S = 0.93$	$\Delta\rho_{\text{max}} = 0.46 \text{ e } \text{\AA}^{-3}$
5265 reflections	$\Delta\rho_{\text{min}} = -0.61 \text{ e } \text{\AA}^{-3}$
316 parameters	
H-atom parameters constrained	

Table 1Selected geometric parameters (\AA , $^\circ$).

Mo1—N1	2.2402 (14)	Mo1—C2	1.965 (2)
Mo1—N2	2.2468 (15)	Mo1—C3	1.963 (2)
Mo1—C1	2.039 (2)	Mo1—C4	2.042 (2)
N1—Mo1—N2	72.82 (5)	N2—Mo1—C4	96.59 (6)
N1—Mo1—C1	97.46 (7)	C1—Mo1—C2	85.93 (7)
N1—Mo1—C2	98.42 (6)	C1—Mo1—C3	88.28 (8)
N1—Mo1—C3	170.97 (7)	C1—Mo1—C4	170.40 (6)
N1—Mo1—C4	89.81 (6)	C2—Mo1—C3	88.90 (8)
N2—Mo1—C1	91.56 (6)	C2—Mo1—C4	86.82 (7)
N2—Mo1—C2	170.52 (7)	C3—Mo1—C4	85.30 (7)
N2—Mo1—C3	100.17 (7)		

H atoms were placed in calculated positions with C—H distances constrained to 0.93 \AA .

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1994); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *SHELXS97* (Sheldrick, 1997); program(s) used to solve structure: *SHELXS97*; program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1990); software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BR1317). Services for accessing these data are described at the back of the journal.

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